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EFFECT OF CHANGING FLY ASH CONTENT, TEMPERATURE, AND CALCIUM CHLORIDE LEVELS ON STRENGTH DEVELOPMENT OF MORTARS AND TIME OF SETTING OF PASTES

by

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This purpose of this project is to quantitatively develop the effect of temperature, fly ash content, and calcium chloride content on time of setting and strength development of pastes and mortars. These findings make it possible to explore the probable effects of increasing the fly ash content of concrete used for revetment mats at the St. Francisville, LA, mat casting facility. The reduction in strength resulting from increasing amount of fly ash proportional to the amount of portland cement could be compensated for by use of 2 percent $CaCl_2$, but this accelerating admixture could not completely compensate for a loss of early strength development that would result from a temperature decrease of 30° F expected from summer to winter.

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Preface

The investigation described in this paper was conducted for the US Army Engineer District, New Orleans (CELMN), by the Concrete Technology Division (CTD) of the Structures Laboratory (SL), US Army Engineer Waterways Experiment Station (WES). The investigation was authorized by DA Form 2544, Intra-Army Order for Reimbursible Services, No. 89-C-0031-5, CELMN, dated 2 January 1990.

The investigation was accomplished under the general supervision of Messrs. Bryant Mather, Chief, SL; James T. Ballard, Assistant Chief, SL; Kennth L. Saucier, Chief, CTD; and under the direct supervision of Toy Poole, Group Leader, Cement and Pozzolan Group. Mel Stegall, Lower Mississippi River Division, conceived the work and designed the experimental approach.

COL Larry B. Fulton, EN, is Commander and Director of WES. Dr. Robert W. Whalin is Technical Director.

Contents

Conversion Factors, Non-SI to SI (Metric) Units of Measurement

Non-SI units of measurement used in this report can be converted to SI (metric) units as follows:

Multiply	By	To Obtain
Fahrenheit degrees	5/9	Celsius degrees or kelvins*
inches	25.4	millimetres
pounds per square inch	0.006894757	megapascals (MPa)
pounds (force)	4.448222	newtons

* To obtain Celsius (C) temperature readings from Fahrenheit (F) readings, use the following formula: $C = (5/9)(F - 32)$. To obtain Kelvin (K) readings, use: $K = (5/9)(F - 32)+273.15$.

EFFECT OF CHANGING FLY ASH CONTENT, TEMPERATURE, AND CALCIUM CHLORIDE LEVELS ON STRENGTH DEVELOPMENT OF MORTARS AND TIME OF SETTING OF PASTES

Introduction

1. The purpose of the work summarized in this report was to investigate the effect of varying fly ash content, calcium chloride ($CaCl_2$) content, and temperature on time of setting and strength development of the portland cement-based cementitious system currently in use for fabrication of revetment mats at the St. Francisville Mat Casting Facility, St. Francisville, Lousiana. Fly ash has been used at a level of 25 percent of the cementitious medium. Consideration is being given to increasing this to 35 percent. This report generates some comparisons in support of this decision. Tests were performed on mortar and paste specimens, therefore, results do not directly indicate concrete properties. Additional data addressing the effect of $CaCl_2$ and temperature on concrete strength development are presented from previously reported work done at WES.

Materials and Methods

2. Cement (LMN 243-89), fly ash (LMN 245C-89), and calcium chloride were obtained from stocks at St. Francisville. Tables 1 and 2 summarize chemical and physical properties of the cement and fly ash, respectively, as determined in standard specification-compliance tests. Standard graded sand from U.S. Silica Company, Ottawa, IL was used for making mortars. Laboratory deionized water was used as mixing water.

3. Time of setting was measured according to ASTM C 191 (ASTM 1989a), except that the water-cement + fly ash ratio was held constant at 0.45 by mass. One determination was made per mixture, except in one instance, as noted in Table 3. The original determination appeared to be out of character with respect to the other results, therefore, a replicate determination was made to verify it.

4. Compressive strength was determined according to ASTM C 109 (ASTM 1989b), except at a constant water-cement + fly ash ratio of 0.45 and storage was in sealed plastic bags. Test ages were 3, 7, 28, and 90 days. Ninety-day

cubes representing all three fly ash levels and 2 percent $CaCl_2$ were lost due to equipment malfunction. Replicate batches were made, cubes cured at 100° (80° conditions no longer available), and compressive-strength determinations made at 28 and 90 days. The relative increase from 28 to 90 days for these cubes was used to estimate the ninety-day strengths for the lost cubes. These data are asterisked in the appropriate tables and figures.

5. Temperature conditions were 50° and 80° F. These were chosen to approximate seasonal changes in temperature. Calcium chloride levels of 0 percent and 2 percent were used. These represent levels used in practice. Levels of fly ash were 0 percent, 25 percent, and 35 percent, by volume of the cementitious medium.

6. Data were analysed by linear regression analysis using the Statistical Analysis System (SAS) software.

7. Data on concrete mixtures were taken from Mather (1965). The cement and fly ash used in these mixtures is unrelated to those used in the experimental program reported here.

Results and Analysis of Data

8. The effects of temperature, calcium chloride content, and fly ash level on initial and final time of setting are illustrated in Figures 1 and 2. Data are summarized in Table 3. Statistical analysis is described in Appendix A. With one exception, the results appear to present a consistent picture that the effects of these variables on time of setting is linear and additive. The exception is in the effect of calcium chloride and temperature on the time of setting of the 0 percent fly ash mixture. Calcium chloride appeared to accelerate both the final and initial time of setting of mixtures without fly ash more at 50° F than expected from a simple extrapolation of its effect on the 25 percent and 35 percent fly ash mixtures.

9. Ignoring the data from the 0 percent mixtures because of this apparent incongruity, the 25 percent and 35 percent mixtures used to quantitatively estimate the effects of amount of fly ash used, temperature, and calcium chloride level. The following equations were developed for times of initial and final setting, respectively, in hours.

$$TOS(i) = 30.28 + (0.13 * \%\ Repl) - (0.33 * Temperature) - (1.86 * \%CaCl_2)$$

$$R^2 = 99\% \quad \text{mean residual} = .4 \text{ hrs}$$

$$TOS(f) = 50.57 + (0.065 * \%Repl) - (0.53 * Temperature) - (1.83 * \%CaCl_2)$$

$$R^2 = 94\% \quad \text{mean residual} = 1.4 \text{ hrs}$$

The coefficient of determination, R^2, indicates the percentage of the total variation in the data that can be accounted for by the regression equation, i.e. a high value of R^2 indicates that the equation is very good at associating changes in time of setting with the independent variables over the range of conditions represented. The mean residual is the average difference (sign ignored) between the observed time of setting and the value predicted by the regression equation for that particular set of conditions. This statistic is not as formal as R^2, but may be more useful in conceptualizing the degree to which the equation explains the observations. The regression constants of these equations indicate the size of the effect of the associated condition on the time of setting. For example, a 10 percent increase in the amount of fly ash results in a 1.3 hour extension of time of initial setting and a 0.65 hour extension of time of final setting. A 10° F temperature increase would result in a 3.3 hour decrease in time of initial setting and a 5.3 hour decrease in time of final setting. Calcium chloride, at 2 percent, decreases time of both initial and final setdting by about 3.7 hours under these temperature and percent replacement conditions. Thus $CaCl_2$, at 2 percent, can compensate for the delay in time of setting caused by a 10 percent increase in fly ash content.

10. Temperature probably has the capacity to have the largest effect, since a 30° temperature decrease (summer to winter) would result in a 9.9 hour extension of time of initial setting and a 15.9 hour extension of time of final set (all other conditions being held constant). Since times of setting of paste specimens are not directly interpretable in terms of times of setting of concrete, these results may be more useful if expressed in terms of percent change from one condition to another. For example, the above data represent a 255 percent and 95 percent increase in times of initial and final setting respectively as compared with the 80° condition. Since the effects of the test conditions appear to be additive the effects of combinations of

conditions can be estimated. For example, the effect of changing from a 25 percent fly ash mixture at 80° (predicted time of initial setting = 7.1 hours) to a 35 percent fly ash mixture at 50° (predicted time of initial setting = 18.3 hours) would be an increase in time of initial setting of 11.2 hours or 157 percent relative to the former condition. Calcium chloride can be used to some effect in compensating for these changes, but over the range of 0 - 2 percent, it cannot completely overcome them.

11. It should be noted that measured values of time of setting are quite method dependent. Therefore, values reported in this work are not directly interpretable in terms of time of setting of concrete. However, relative changes in time of setting, as reported in this work, should reflect changes to be expected in times of setting of concrete.

12. Patterns of strength development are illustrated in Figures 3 and 4. Statistical analysis is described in Appendix B. The following regression equations were developed to relate strength to percent replacement, temperature and calcium chloride level. Unlike the time of setting data, these analyses include the 0 percent fly ash (fa) level.

$$\text{3-day PSI} = 40.7 - (30.0*\%\text{fly ash}) + (47.3*\text{Temp}) + (444.2*\%CaCl_2)$$
$$R^2=99\% \quad \text{mean residual} = 90 \text{ psi}$$

$$\text{7-day PSI} = 2070 - (30.1*\%\text{fly ash}) + (31.9*\text{Temp}) + (380.0*\%CaCl_2)$$
$$R^2=91\% \quad \text{mean residual} = 196 \text{ psi}$$

$$\text{28-day PSI} = 3938 - (30.1*\%\text{fly ash}) + (28.9*\text{Temp}) + (268.3*\%CaCl_2)$$
$$R^2=83\% \quad \text{mean residual} = 252 \text{ psi}$$

$$\text{90-day PSI} = 7992 - (22.7*\%\text{fly ash}) + (4.4*\text{Temp}) + (304.9*\%CaCl_2)$$
$$R^2=66\% \quad \text{mean residual} = 249 \text{ psi}$$

13. As in the data on time of setting, these equations are useful for estimating expected changes in strength for various combinations of test conditions. For example, for an increase in fly ash level of 10 percent, but with a constant temperature and calcium chloride levels, an expected strength decrease of 300 psi would occur at 3 days. This effect appears to be about

the same at 7 and 28 days. Two percent $CaCl_2$ can overcome this loss in early strength.

14. The equation representing the 3-day test age appears to be quite accurate in associating strength with a given change in fly ash level, temperature, and calcium chloride level. This accuracy tends to decrease with increasing test age, as indicated by the decreasing R^2. This may be due to a lack of simple additivity of the effects of each of the independent variables.

15. As in the time-of-setting analysis, temperature has the potential to have the greatest effect on strength because of the range of variation it is likely to exhibit. According to the above equation for 3-day strength, a 30° temperature drop would cause a reduction of 1419 psi. This could not be completely overcome by addition of 2 percent $CaCl_2$. Use of levels much higher than 2 percent would probably not be desirable because of a tendency for accelerators to give diminishing returns with increasing dosage, and in some cases an actual reduction in strength might occur (Ramachandran 1984). Also the Corps of Engineers generally (CW 03301, Sec 6.3.2) limits the use of calcium chloride to 1 percent by mass of the cement.

16. These equations also indicate that the strength-enhancing effect of temperature decreases with time, as indicated by the smaller regression constants at later ages. The accelerating effect of $CaCl_2$ also diminishes with time from 3 to 28 days, but has a stronger effect at 90 days, as indicated by the size of regression coefficients at these ages. The 90-day coefficient may be an anomaly that resulted from the strength estimation that was done at this age as a result of the equipment malfunction. In general, factors that accelerate cement hydration at early ages tend to result in slower hydration at later ages. For $CaCl_2$ to actually have as strong effect at 90 days as at 3 days would be contrary to this generality. Examination of the data generated in some concrete mixtures is enlightening, as described below.

17. Examination of the data on concretes (Table 4) supports the strength-development data obtained in mortars in that the addition of $CaCl_2$ largely compensates for the strength lost due to a relatively large proportion of fly ash in the mixture, but only partially overcomes the inhibition in strength gain resulting from a 30°F temperature decrease. These data represent cooler curing conditions than used with the mortar specimens, but the range of temperatures is about the same (30°F). These data did not reflect

the strong effect of $CaCl_2$ at 90 days that was observed in the mortar data. That pattern in the mortar data was probably due to an anomaly.

Conclusions

18. Except for the 0 percent fly ash mixtures, time of setting appears to be affected by fly ash content, temperature, and $CaCl_2$ level in an approximately additive way, over the range of conditions represented. This allows the use of multiple linear regression to generate equations that can be used as a tool to estimate the results of hypothetical changes in conditions.

19. The proposed increase in percent fly ash by 10 percent results in a delay of time of setting that can be overcome by use of 2 percent $CaCl_2$. A 30° F decrease in temperature causes a delay in time of setting that cannot be compensated by 2 percent $CaCl_2$.

20. Compressive strength at a given age also appears to be affected by fly ash content, temperature, and $CaCl_2$ in an approximately additive way, particularly at early ages.

21. The proposed increase in percent fly ash by 10 percent causes a decrease in early strength that can be compensated by addition of 2 percent $CaCl_2$, but a decrease in temperature of 30° causes a strength reduction that cannot be so compensated.

22. Temperature causes an acceleration in early strength but strengths at later ages are depressed relative to unaccelerated mixtures. $CaCl_2$ causes an acceleration in early strength that persists at least through 90 days.

<u>References</u>

American Society for Testing and Materials. 1989a. Annual Book of Standards. "Standard Test Method for Time of Setting of Hydraulic Cement by Vicat Needle." Designation: C 191-82, Philadelphia, PA.

__________. 1989b. Anual Book of Standards. "Standard Test Method for Compressive Strength of Hydraulic Cement Mortars (Using 2-in. or 50-mm Cube Specimens)." Designation: C 109-88, Philadelphia, PA.

Mather, B. 1965. "Investigation of Cement-Replacement Materials, Compressive Strength Development of 193 Concrete Mixtures During 10 Years of Moist Curing (Phase A), MP 6-123, USAE Waterways Experiment Station, Vicksburg, MS.

Ramachandran, V.S. 1984. <u>Concrete Admixtures Handbook</u>, Noyes Publications, Park Ridge, New Jersey, USA.

Table 1

Physical and chemical properties of portland cement tested for compliance with Type I specifications (ASTM C 150)

Company: River Cement
Location: Festus, Missouri
Specification: ASTM C 150, I,LA,FS
Project: St. Francisville Mat Casting

Test Report No.: LMN-243-89
Program: Single Sample
CTD No.:
Date Sampled: 24 October 1989

Chemical Analysis	Result	Spec Limits Type I
SiO_2, %	20.8	-
Al_2O_3, %	4.3	-
Fe_2O_3, %	3.6	-
CaO, %	62.7	-
MgO, %	3.2	6.0 max
SO_3, %	2.6	3.0 max
Loss on ignition, %	1.2	3.0 max
Insoluble residue, %	0.22	0.75 max
Na_2O, %	0.04	-
K_2O, %	0.44	-
Alkalies-total as Na_2O,%	0.33	0.60 max
TiO_2, %	0.27	-
P_2O_5, %	0.28	-
C_3A, %	7	15 max
C_3S, %	52	-
C_2S, %	20	-
C_4AF, %	11	-

Physical Tests	Result	Spec Limits Type I
Heat of hydration, 7-day, cal/g	-	-
Surface area, m^2/kg (air permeability)	420	280 min
Autoclave expansion, %	0.00	0.80 max
Initial set, min. (Gillmore)	205	60 min
Final set, min. (Gillmore)	310	600 max
Air content, %	10	12 max
Compressive strength, 3-day, psi	2880	1800 min
Compressive strength, 7-day, psi	3890	2800 min
False set (final penetration), %	79	50 min

Table 2

Physical and chemical properties of pozzolan tested for compliance with Class C specifications (ASTM C 618).

Company: Bayou Ash
Location: New Roads, Louisiana
Specification: ASTM C 618, Class C
Project: St. Francisville Mat Casting

Test Report No.: LMN-245C-89
Program: Single Sample
CTD No.:
Date Sampled: 24 October 1989

Chemical Analysis	Result	Spec Limits Class C
SiO_2, %	41.2	-
Al_2O_3, %	20.5	-
Fe_2O_3, %	7.2	-
Sum, %	68.8	50.0 min
MgO, %	5.6	-
SO_3, %	1.9	5.0 max
Moisture content, %	0.3	3.0 max
Loss on ignition, %	1.3	6.0 max
Available alkalies (28-day), %	0.8	1.5 max

Physical Tests	Result	Spec Limits Class C
Fineness (45 micrometre), % retained	19	34 max
Fineness variation, %	5	5 max
Water requirement, %	92	105 max
Density, Mg/m^3	2.62	-
Density variation, %	4	5 max
Autoclave expansion, %	-0.08	0.80 max
Pozzolanic activity w/cement (28-day), %	108	75 min

Laboratory cement used: River Cement, Festus, Missouri
Laboratory lime used: Chemstone

Table 3
Time-of-set and compressive-strength data

TEMP (° F)	$CaCl_2$ %	Repl %	Initial Set (hr)	Final Set (hr)	3-Day PSI	7-Day PSI	28-Day PSI	90-Day PSI
50	0	0	15.2	22.8	2390	3750	5430	6850
50	0	25	16.6	24.4	1710	3250	5010	6400
50	0	35	18.7	24.9	1230	2280	4020	6290
50	2	0	4.3[1]	6.7[1]	3350	4490	6040	7080
50	2	25	13.8	23.8	2590	3670	5220	6790
50	2	35	15.0	24.0	2220	3220	4580	5850
80	0	0	5.9	9.3	3670	4260	5720	6210
80	0	25	8.2	11.6	3200	3990	5650	5950
80	0	35	8.4	11.8	2880	3720	5460	6130
80	2	0	2.3	4.1	4750	5420	6970	8050[2]
80	2	25	3.2	4.3	3980	4780	6080	7071[2]
80	2	35	5.0	6.0	3520	4230	5620	6648[2]

[1]Mean of duplicate determinations
[2]Values estimated from other test results

Table 4. Effect of $CaCl_2$, temperature, and fly ash replacement on compressive strength of concretes.

cement + fly ash lbs/yd	% fly ash	w/c	3 day	7 day	28 day	90 day	365 day	5 year	10 yr
With calcium chloride, mixed at 73 deg F, specimens cured at 73 deg F									
514	0	0.5	2860	3430	4810	5520	6340	7020	6430
344	0	0.8	1020	1375	2080	2660	2930	3190	2980
289	45	0.8	360	560	1130	1710	2520	2820	2810
With calcium chloride, mixed at 40 deg F, specimens cured at 73 deg F									
507	0	0.5	3070	3900	5340	6620	7500	7890	8070
345	0	0.8	1080	1460	2200	2690	3220	3470	3350
298	45	0.8	350	590	1210	1770	2610	3010	2910
With calcium chloride, mixed at 40 deg F, specimens cured at 40 deg F									
509	0	0.5	1820	3020	4790	6210	7200	8380	8070
349	0	0.8	610	1170	1900	2650	3550	3560	3730
301	45	0.8	210	330	570	1180	2770	3250	2880
No Calcium Chloride									
532	0	0.5	1860	3190	4440	5470	6070	7060	7250
351	0	0.8	820	1290	1990	2620	2850	2970	3380
425	45	0.5	1080	1530	2810	4300	5240	6140	6070
296	45	0.8	350	530	1130	1720	2450	2770	2750

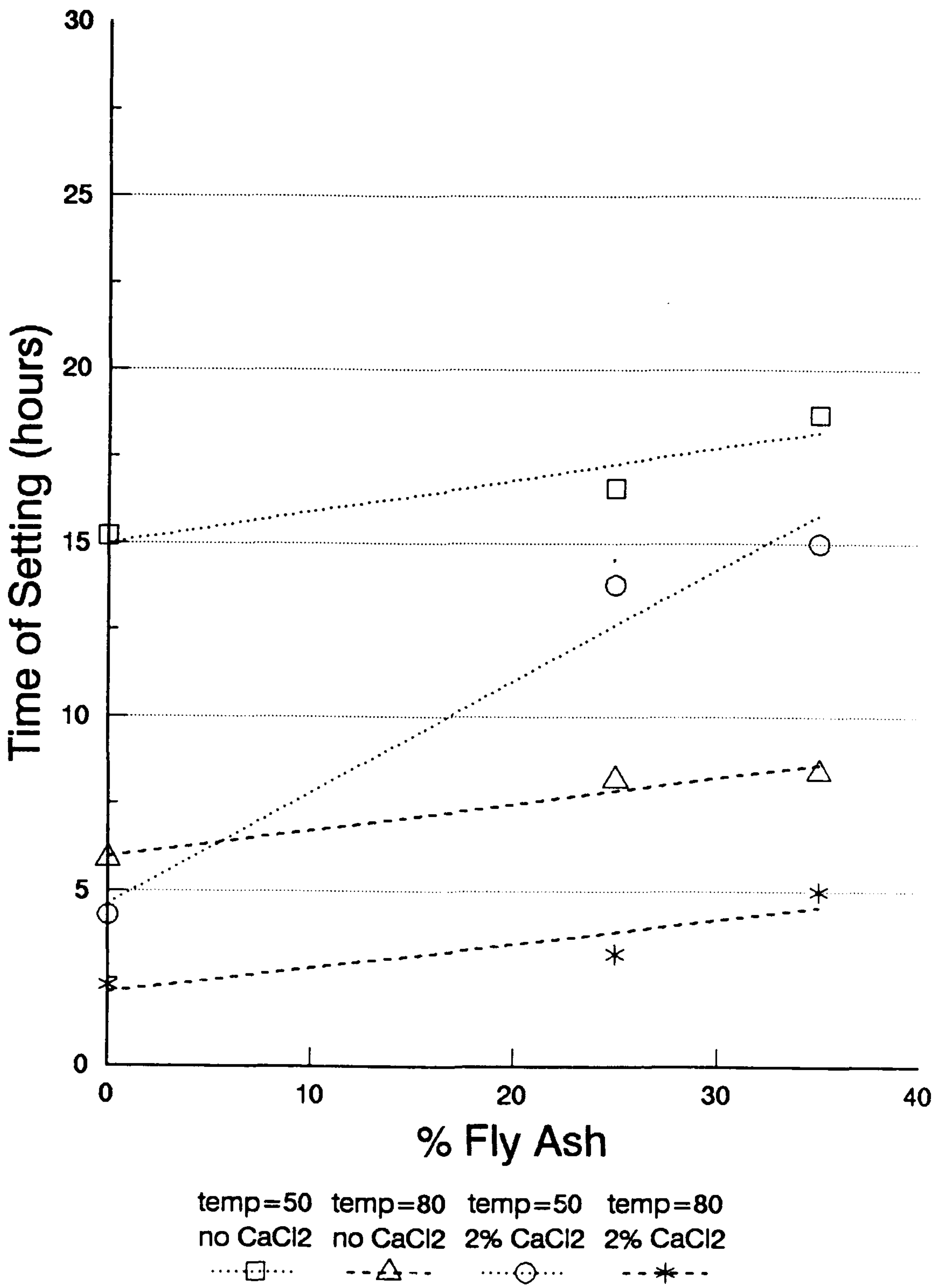

Figure 1. Initial time of setting versus percent fly ash at 50° and 80° F, with and without $CaCl_2$

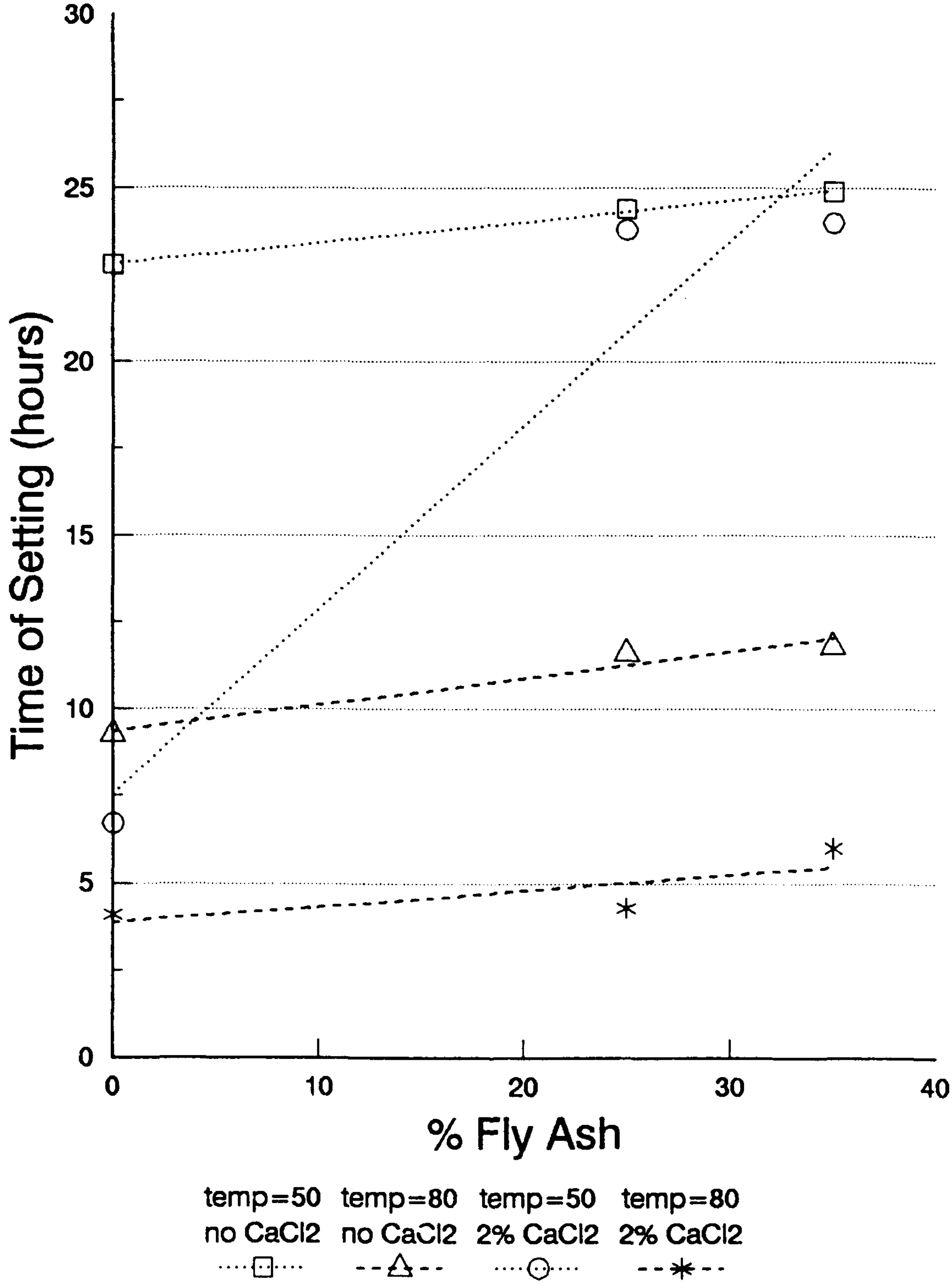

Figure 2. Final time of setting versus percent fly ash at 50° and 80° F, with and without $CaCl_2$

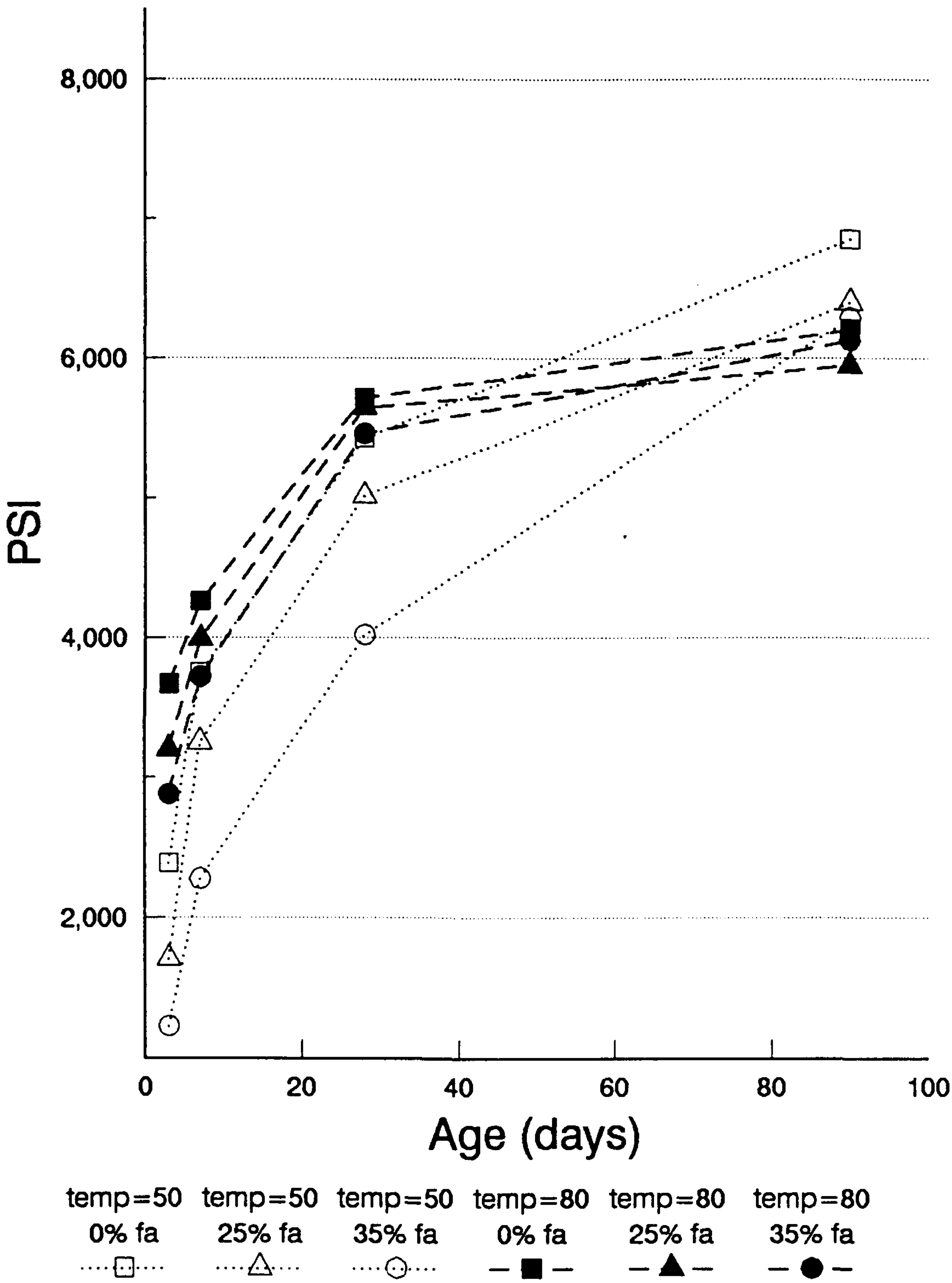

Figure 3. Compressive strength versus age at 50° and 80° F; 0, 25, and 35 percent fly ash; no $CaCl_2$

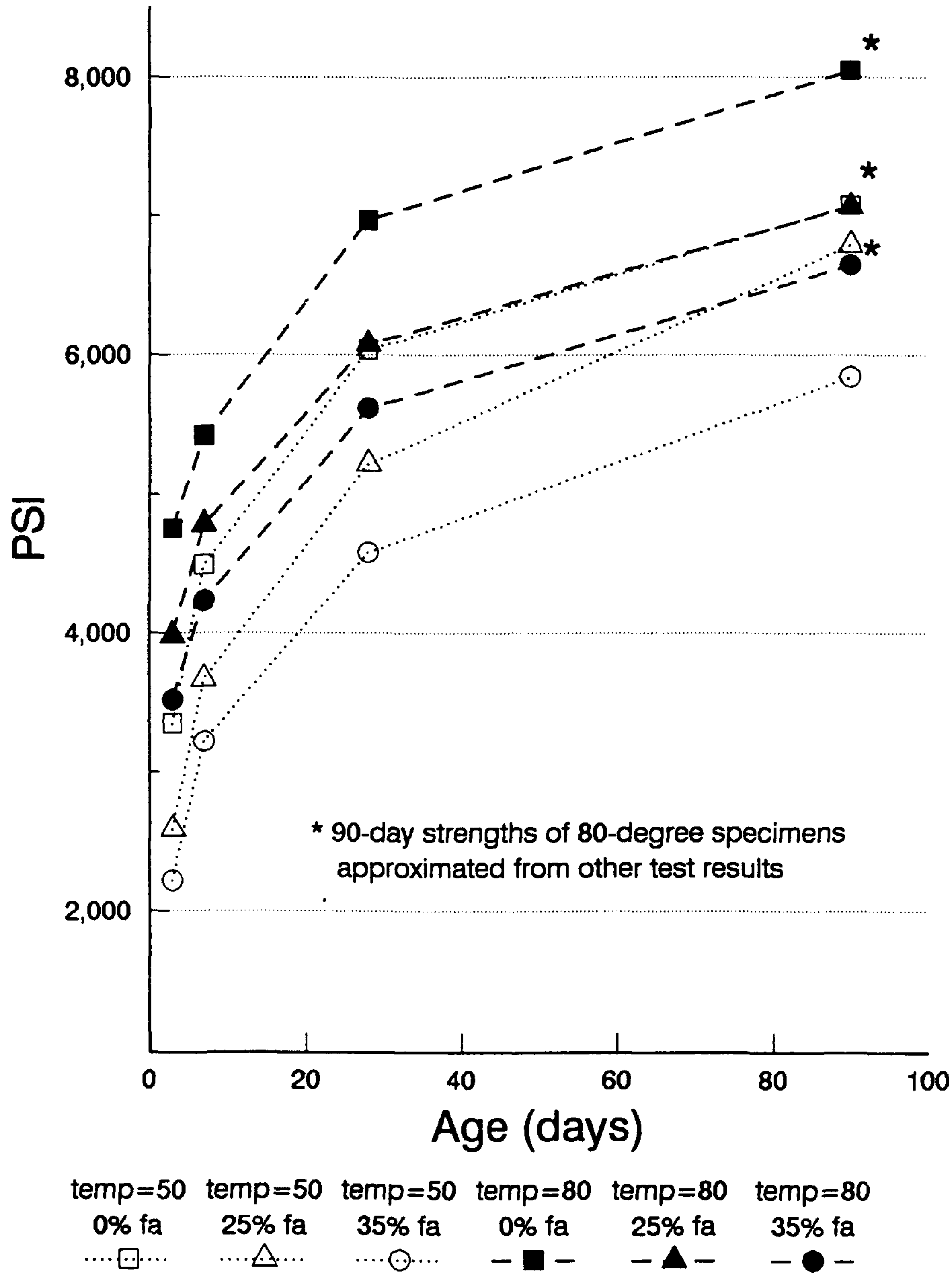

Figure 4. Compressive strength versus age at 50° and 80° F; 0, 25, and 35 percent fly ash; 2 percent $CaCl_2$

Appendix A. Statistical Analysis of Time-of-Setting Data.

```
DATA ONE;
DO PCT_RPL=0,25,35;
DO CACL2=0,2;
DO TEMP=50,80;
DO TYPE="I","F";
INPUT TOS @; OUTPUT;
END;
END;
END;
END;
CARDS;
15.2 22.8 5.9 9.25
4.3 6.7 2.3 4.1
16.6 24.4 8.2 11.6
13.8 23.8 3.2 4.3
18.7 24.9 8.4 11.8
15.0 24.0 5.0 6.0
;
PROC SORT; BY TYPE;
TITLE "ALL DATA";
PROC REG; BY TYPE;
MODEL TOS=PCT_RPL TEMP CACL2/ P;
DATA TWO; SET ONE;
IF PCT_RPL=0 THEN DELETE;
TITLE "0% DATA DELETED";
PROC REG; BY TYPE;
MODEL TOS=PCT_RPL TEMP CACL2/ P;
RUN;
```

ALL DATA

FINAL SET

Model: MODEL1
Dependent Variable: TOS

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Value	Prob>F
Model	3	717.25824	239.08608	15.540	0.0011
Error	8	123.08405	15.38551		
C Total	11	840.34229			

Root MSE	3.92244	R-square	0.8535
Dep Mean	14.47083	Adj R-sq	0.7986
C.V.	27.10581		

Parameter Estimates

Variable	DF	Parameter Estimate	Standard Error	T for H0: Parameter=0	Prob > |T|
INTERCEP	1	42.615491	5.38578882	7.913	0.0001
PCT_FA	1	0.178462	0.07692531	2.320	0.0489
TEMP	1	-0.441944	0.07548732	-5.855	0.0004
CACL2	1	-2.987500	1.13230984	-2.638	0.0298

Obs	Dep Var TOS	Predict Value	Residual
1	22.8000	20.5183	2.2817
2	9.2500	7.2599	1.9901
3	6.7000	14.5433	-7.8433
4	4.1000	1.2849	2.8151
5	24.4000	24.9798	-0.5798
6	11.6000	11.7215	-0.1215
7	23.8000	19.0048	4.7952
8	4.3000	5.7465	-1.4465
9	24.9000	26.7644	-1.8644
10	11.8000	13.5061	-1.7061
11	24.0000	20.7894	3.2106
12	6.0000	7.5311	-1.5311

Sum of Residuals	2.930989E-14
Sum of Squared Residuals	123.0841
Predicted Resid SS (Press)	310.8385

ALL DATA

------------------------------- INITIAL SET --------------------------------

Model: MODEL1
Dependent Variable: TOS

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Value	Prob>F
Model	3	335.65574	111.88525	25.764	0.0002
Error	8	34.74093	4.34262		
C Total	11	370.39667			

Root MSE	2.08389	R-square	0.9062
Dep Mean	9.71667	Adj R-sq	0.8710
C.V.	21.44660		

Parameter Estimates

Variable	DF	Parameter Estimate	Standard Error	T for H0: Parameter=0	Prob > |T|
INTERCEP	1	27.658120	2.86133785	9.666	0.0001
PCT_FA	1	0.139038	0.04086853	3.402	0.0093
TEMP	1	-0.281111	0.04010457	-7.009	0.0001
CACL2	1	-2.450000	0.60156852	-4.073	0.0036

Obs	Dep Var TOS	Predict Value	Residual
1	15.2000	13.6026	1.5974
2	5.9000	5.1692	0.7308
3	4.3000	8.7026	-4.4026
4	2.3000	0.2692	2.0308
5	16.6000	17.0785	-0.4785
6	8.2000	8.6452	-0.4452
7	13.8000	12.1785	1.6215
8	3.2000	3.7452	-0.5452
9	18.7000	18.4689	0.2311
10	8.4000	10.0356	-1.6356
11	15.0000	13.5689	1.4311
12	5.0000	5.1356	-0.1356

Sum of Residuals	9.769963E-15
Sum of Squared Residuals	34.7409
Predicted Resid SS (Press)	91.8349

-------------------------------- FINAL SET --------------------------------

Model: MODEL1
Dependent Variable: TOS

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Value	Prob>F
Model	3	529.93500	176.64500	40.181	0.0019
Error	4	17.58500	4.39625		
C Total	7	547.52000			

Root MSE	2.09672	R-square	0.9679
Dep Mean	16.35000	Adj R-sq	0.9438
C.V.	12.82400		

Parameter Estimates

Variable	DF	Parameter Estimate	Standard Error	T for H0: Parameter=0	Prob > |T|
INTERCEP	1	50.566667	5.58580014	9.053	0.0008
PCT_FA	1	0.065000	0.14826075	0.438	0.6837
TEMP	1	-0.528333	0.04942025	-10.691	0.0004
CACL2	1	-1.825000	0.74130375	-2.462	0.0696

Obs	Dep Var TOS	Predict Value	Residual
1	24.4000	25.7750	-1.3750
2	11.6000	9.9250	1.6750
3	23.8000	22.1250	1.6750
4	4.3000	6.2750	-1.9750
5	24.9000	26.4250	-1.5250
6	11.8000	10.5750	1.2250
7	24.0000	22.7750	1.2250
8	6.0000	6.9250	-0.9250

Sum of Residuals	-5.59552E-14
Sum of Squared Residuals	17.5850
Predicted Resid SS (Press)	70.3400

---------------------------------- INITIAL SET ----------------------------------

Model: MODEL1
Dependent Variable: TOS

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Value	Prob>F
Model	3	224.32375	74.77458	198.736	0.0001
Error	4	1.50500	0.37625		
C Total	7	225.82875			

Root MSE	0.61339	R-square	0.9933
Dep Mean	11.11250	Adj R-sq	0.9883
C.V.	5.51984		

Parameter Estimates

Variable	DF	Parameter Estimate	Standard Error	T for H0: Parameter=0	Prob > |T|
INTERCEP	1	30.287500	1.63411440	18.535	0.0001
PCT_FA	1	0.132500	0.04337338	3.055	0.0378
TEMP	1	-0.327500	0.01445779	-22.652	0.0001
CACL2	1	-1.862500	0.21686689	-8.588	0.0010

Obs	Dep Var TOS	Predict Value	Residual
1	16.6000	17.2250	-0.6250
2	8.2000	7.4000	0.8000
3	13.8000	13.5000	0.3000
4	3.2000	3.6750	-0.4750
5	18.7000	18.5500	0.1500
6	8.4000	8.7250	-0.3250
7	15.0000	14.8250	0.1750
8	5.0000	5.0000	-178E-17

Sum of Residuals	-3.9968E-14
Sum of Squared Residuals	1.5050
Predicted Resid SS (Press)	6.0200

Appendix B. Statistical analysis of compressive strength data.

```
DATA ONE;
DO PCT_RPL=0,25,35;
DO CACL2=0,2;
DO TEMP=50,80;
DO AGE=3,7,28,90;
INPUT PSI @; OUTPUT;
END;
END;
END;
END;
CARDS;
2390 3750 5430 6850
3670 4260 5720 6210
3350 4490 6040 7080
4750 5420 6970 8050
1710 3250 5010 6400
3200 3990 5650 5950
2590 3670 5220 6790
3980 4780 6080 7071
1230 2280 4020 6290
2880 3720 5460 6130
2220 3220 4580 5850
3520 4230 5620 6648
;
PROC SORT; BY AGE;
PROC PRINT;
TITLE "ALL DATA";
PROC REG; BY AGE;
MODEL PSI=PCT_RPL TEMP CACL2/ P;
PROC REG;
MODEL PSI=AGE PCT_RPL TEMP CACL2/ P;
RUN;
```

---------------------------------- AGE=3 ----------------------------------

Model: MODEL1
Dependent Variable: PSI

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Value	Prob>F
Model	3	10745417.628	3581805.8761	293.568	0.0001
Error	8	97607.37179	12200.92147		
C Total	11	10843025.000			

Root MSE	110.45778	R-square	0.9910
Dep Mean	2957.50000	Adj R-sq	0.9876
C.V.	3.73484		

Parameter Estimates

Variable	DF	Parameter Estimate	Standard Error	T for H0: Parameter=0	Prob > |T|
INTERCEP	1	40.662393	151.66652377	0.268	0.7954
PCT_FA	1	-30.019231	2.16625532	-13.858	0.0001
TEMP	1	47.277778	2.12576099	22.240	0.0001
CACL2	1	444.166667	31.88641492	13.930	0.0001

Obs	Dep Var PSI	Predict Value	Residual
1	2390.0	2404.6	-14.5513
2	3670.0	3822.9	-152.9
3	3350.0	3292.9	57.1154
4	4750.0	4711.2	38.7821
5	1710.0	1654.1	55.9295
6	3200.0	3072.4	127.6
7	2590.0	2542.4	47.5962
8	3980.0	3960.7	19.2628
9	1230.0	1353.9	-123.9
10	2880.0	2772.2	107.8
11	2220.0	2242.2	-22.2115
12	3520.0	3660.5	-140.5

Sum of Residuals	4.092726E-12
Sum of Squared Residuals	97607.3718
Predicted Resid SS (Press)	227242.4998

--------------------------------- AGE=7 ---------------------------------

Model: MODEL1
Dependent Variable: PSI

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Value	Prob>F
Model	3	6830448.7179	2276816.2393	39.552	0.0001
Error	8	460517.94872	57564.74359		
C Total	11	7290966.6667			

Root MSE	239.92654	R-square	0.9368
Dep Mean	3921.66667	Adj R-sq	0.9132
C.V.	6.11797		

Parameter Estimates

Variable	DF	Parameter Estimate	Standard Error	T for H0: Parameter=0	Prob > |T|
INTERCEP	1	2070.427350	329.43649147	6.285	0.0002
PCT_FA	1	-30.076923	4.70534653	-6.392	0.0002
TEMP	1	31.888889	4.61738837	6.906	0.0001
CACL2	1	380.000000	69.26082562	5.487	0.0006

Obs	Dep Var PSI	Predict Value	Residual
1	3750.0	3664.9	85.1282
2	4260.0	4621.5	-361.5
3	4490.0	4424.9	65.1282
4	5420.0	5381.5	38.4615
5	3250.0	2912.9	337.1
6	3990.0	3869.6	120.4
7	3670.0	3672.9	-2.9487
8	4780.0	4629.6	150.4
9	2280.0	2612.2	-332.2
10	3720.0	3568.8	151.2
11	3220.0	3372.2	-152.2
12	4230.0	4328.8	-98.8

Sum of Residuals	4.547474E-13
Sum of Squared Residuals	460517.9487
Predicted Resid SS (Press)	1056613.1438

---------------------------------- AGE=28 ----------------------------------

Model: MODEL1
Dependent Variable: PSI

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Value	Prob>F
Model	3	5469382.0513	1823127.3504	19.108	0.0005
Error	8	763284.61538	95410.57692		
C Total	11	6232666.6667			

Root MSE	308.88603	R-square	0.8775
Dep Mean	5483.33333	Adj R-sq	0.8316
C.V.	5.63318		

Parameter Estimates

Variable	DF	Parameter Estimate	Standard Error	T for H0: Parameter=0	Prob > |T|
INTERCEP	1	3938.760684	424.12285646	9.287	0.0001
PCT_FA	1	-30.076923	6.05775336	-4.965	0.0011
TEMP	1	28.888889	5.94451434	4.860	0.0013
CACL2	1	268.333333	89.16771507	3.009	0.0168

Obs	Dep Var PSI	Predict Value	Residual
1	5430.0	5383.2	46.7949
2	5720.0	6249.9	-529.9
3	6040.0	5919.9	120.1
4	6970.0	6786.5	183.5
5	5010.0	4631.3	378.7
6	5650.0	5497.9	152.1
7	5220.0	5167.9	52.0513
8	6080.0	6034.6	45.3846
9	4020.0	4330.5	-310.5
10	5460.0	5197.2	262.8
11	4580.0	4867.2	-287.2
12	5620.0	5733.8	-113.8

Sum of Residuals 0
Sum of Squared Residuals 763284.6154
Predicted Resid SS (Press) 1836759.8806

------------------------------------ AGE=90 ------------------------------------

Model: MODEL1
Dependent Variable: PSI

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Value	Prob>F
Model	3	2506601.9455	835533.98184	4.071	0.0499
Error	8	1641962.9712	205245.37139		
C Total	11	4148564.9167			

Root MSE	453.04014	R-square	0.6042
Dep Mean	6609.91667	Adj R-sq	0.4558
C.V.	6.85395		

Parameter Estimates

Variable	DF	Parameter Estimate	Standard Error	T for H0: Parameter=0	Prob > |T|
INTERCEP	1	6470.126068	622.05688704	10.401	0.0001
PCT_FA	1	-22.682692	8.88484820	-2.553	0.0340
TEMP	1	4.438889	8.71876162	0.509	0.6244
CACL2	1	304.916667	130.78142433	2.331	0.0480

Obs	Dep Var PSI	Predict Value	Residual
1	6850.0	6692.1	157.9
2	6210.0	6825.2	-615.2
3	7080.0	7301.9	-221.9
4	8050.0	7435.1	614.9
5	6400.0	6125.0	275.0
6	5950.0	6258.2	-308.2
7	6790.0	6734.8	55.1633
8	7071.0	6868.0	203.0
9	6290.0	5898.2	391.8
10	6130.0	6031.3	98.7
11	5850.0	6508.0	-658.0
12	6648.0	6641.2	6.8237

Sum of Residuals	2.000888E-11
Sum of Squared Residuals	1641962.9712
Predicted Resid SS (Press)	4084315.2910

Model: MODEL1
Dependent Variable: PSI

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Value	Prob>F
Model	4	100813741.62	25203435.404	48.265	0.0001
Error	43	22454320.862	522193.50841		
C Total	47	123268062.48			

Root MSE	722.62958	R-square	0.8178
Dep Mean	4743.10417	Adj R-sq	0.8009
C.V.	15.23537		

Parameter Estimates

Variable	DF	Parameter Estimate	Standard Error	T for H0: Parameter=0	Prob > |T|
INTERCEP	1	1957.017373	505.29375758	3.873	0.0004
AGE	1	36.655523	2.99663196	12.232	0.0001
PCT_FA	1	-28.213942	7.08596601	-3.982	0.0003
TEMP	1	28.123611	6.95350637	4.045	0.0002
CACL2	1	349.354167	104.30259549	3.349	0.0017

Obs	Dep Var PSI	Predict Value	Residual
1	2390.0	3473.2	-1083.2
2	3670.0	4316.9	-646.9
3	3350.0	4171.9	-821.9
4	4750.0	5015.6	-265.6
5	1710.0	2767.8	-1057.8
6	3200.0	3611.5	-411.5
7	2590.0	3466.5	-876.5
8	3980.0	4310.2	-330.2
9	1230.0	2485.7	-1255.7
10	2880.0	3329.4	-449.4
11	2220.0	3184.4	-964.4
12	3520.0	4028.1	-508.1
13	3750.0	3619.8	130.2
14	4260.0	4463.5	-203.5
15	4490.0	4318.5	171.5
16	5420.0	5162.2	257.8
17	3250.0	2914.4	335.6
18	3990.0	3758.1	231.9

Obs	Dep Var PSI	Predict Value	Residual
19	3670.0	3613.1	56.8536
20	4780.0	4456.9	323.1
21	2280.0	2632.3	-352.3
22	3720.0	3476.0	244.0
23	3220.0	3331.0	-111.0
24	4230.0	4174.7	55.2847
25	5430.0	4389.6	1040.4
26	5720.0	5233.3	486.7
27	6040.0	5088.3	951.7
28	6970.0	5932.0	1038.0
29	5010.0	3684.2	1325.8
30	5650.0	4527.9	1122.1
31	5220.0	4382.9	837.1
32	6080.0	5226.6	853.4
33	4020.0	3402.1	617.9
34	5460.0	4245.8	1214.2
35	4580.0	4100.8	479.2
36	5620.0	4944.5	675.5
37	6850.0	6662.2	187.8
38	6210.0	7505.9	-1295.9
39	7080.0	7360.9	-280.9
40	8050.0	8204.6	-154.6
41	6400.0	5956.8	443.2
42	5950.0	6800.6	-850.6
43	6790.0	6655.6	134.4
44	7071.0	7499.3	-428.3
45	6290.0	5674.7	615.3
46	6130.0	6518.4	-388.4
47	5850.0	6373.4	-523.4
48	6648.0	7217.1	-569.1

Sum of Residuals	5.456968E-12
Sum of Squared Residuals	22454320.862
Predicted Resid SS (Press)	27718651.958